

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

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Title: Animal Tissue Analysis		
Revision: 5	Replaces: 01/01/06	Effective: 03/01/06

1. Purpose:

To provide standard procedures for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS Pesticide Data Program (PDP) animal tissue samples.

2. Scope:

This standard operating procedure (SOP) shall be followed by the analytical laboratory conducting pesticide residue studies for animal tissues, the USDA, AMS National Science Laboratory {NSL (US2)}. Sampling shall be performed by designated USDA, Food Safety and Inspection Service (FSIS) inspectors under established sampling protocols. All samples shall be shipped frozen to NSL. Each sample shall be comprised of designated tissues (e.g., adipose/liver/muscle). Any reportable residue results shall initiate immediate action reporting to the USDA, AMS Monitoring Programs Office (MPO); the official USDA, FSIS designee(s) and the FSIS residue mailbox (see section 6.5.a).

3. Outline of Procedures:

- 6.1 Sample Receipt
- 6.2 Sample Preparation
- 6.3 Sample Storage
- 6.4 Sample Analysis
- 6.5 Data Reporting
- 6.6 Sample Disposal

Attachment 1, FSIS Unified Sampling Form, Form 10,210-3

Attachment 2, Reportable Residue Form for Animal Tissues

4. References:

- USDA, AMS/USDA, FSIS Conference Call, January 31, 2006
 - USDA, AMS/ USDA, FSIS Planning Meeting, September 7, 2005
 - USDA, AMS/ USDA, FSIS Planning Meeting, October 19, 2004
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- USDA, AMS/EPA, HED Planning Meeting, October 14, 2004
- USDA, AMS/EPA PDP Planning Meeting, January 30, 2002
- Memorandum, Martha Lamont, PDP Technical Director, to Ed Zager, Chief, EPA/HED, August 24, 2000
- USDA/AMS, USDA OPMP, EPA Planning Meeting, August 23, 2000
- Memorandum, OPs in Meat and Poultry, Martha Lamont, EPA/HED, June 8, 1998
- Pesticide Chemical News Guide

5. Summary:

No site shall be sampled more than once on a single day. Each poultry sample shall be a frozen composite of six birds from the same flock and each beef or pork sample shall be collected from a single animal. Each sample shall be comprised of approximately one pound of each of the designated tissues (e.g., adipose/liver/muscle). Each tissue shall be analyzed for identified compounds (refer to applicable SOP PDP-QC-13 addendum). If a sample contains a residue concentration \$the level of quantitation (LOQ), a reportable residue has occurred and shall be reported immediately to MPO; the USDA, FSIS designee(s); and the FSIS residue mailbox (see section 6.5.a) using the PDP Reportable Residue Form for Animal Tissues (Attachment 2).

6. Procedure:

This SOP represents minimum PDP requirements for the receipt, preparation, storage, analysis, reporting, and disposal of PDP animal tissue samples and is presented as a general guideline. The analytical laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in the laboratory.

6.1 Sample Receipt

6.1.a Quarterly sampling plans designating sampling dates and sites shall be prepared by USDA, FSIS and provided to the analytical laboratory. Samples shall be collected on Mondays, except when Monday is a holiday. In this case, samples shall be collected on Tuesday. Samples shall be frozen then shipped by overnight courier to the laboratory on the day after sample collection. If a sample is not received during the expected timeframe, the laboratory shall immediately contact the

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PDP Sampling Manager; the USDA, FSIS designee(s); and the USDA, FSIS residue mailbox(see section 6.5.a). Samples that are not received by Wednesday at the laboratory will be held and analyzed with the next's week samples.

6.1.b Each poultry sample sent shall represent a composite of tissue from six birds in the same flock; each beef and pork sample shall be collected from a single animal. Each sample shall be comprised of approximately one pound of each of the designated tissues (e.g., adipose/liver/muscle).

6.1.c Samples shall be shipped frozen. Samples that have not completely thawed in transit (still cold to the touch) may be homogenized and stored until time of analysis. Samples and homogenates shall be stored in accordance with the requirements specified in SOP PDP-LABOP-01, sections 5.2.f through 5.2.4.

6.1.d Those samples, or portions thereof, received in a damaged (e.g., warm to the touch, spoiled, or leaking) condition shall be discarded and not analyzed. Condition and disposal shall be recorded on all applicable documentation, for example, the appropriate USDA, FSIS Unified Sampling Form 10,210-3. If a sample must be discarded, the laboratory shall immediately notify MPO; the USDA, FSIS designee(s); and the FSIS residue mailbox (see section 6.5.a).

6.1.e Each sample shall be accompanied by a Unified Sampling Form (Attachment 1) completed by the USDA, FSIS inspector collecting the sample. The laboratory shall assign the PDP sample identification number. This number shall be a concatenation of the state, sampling date, sampling site, tissue, and analytical laboratory. This information is located in the following blocks of the USDA, FSIS Unified Sampling Form: state (6), sampling date (19), sampling site (3) and tissue (12).

NOTE: The USDA, FSIS site code is a seven-digit code which must be cross-referenced with the PDP Site Code List.

Poultry, beef, and pork tissues shall have the following commodity codes, respectively: adipose (PA, BA, KA), liver (PL, BL), and muscle {PM (poultry thigh = PT and poultry breast tissue = PR, BM, KM)}. The laboratory designation is US2.

Example: For a poultry sample collected at site 3002 in Wilkesboro, North Carolina, on April 15, 2000, the following sample identification would be entered on the appropriate PDP SIF:

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adipose (NC-00-04-15-3002-PA-US2)

liver (NC-00-04-15-3002-PL-US2)

muscle (NC-00-04-15-3002-PM-US2)

6.1.f The laboratory shall maintain a log of samples received. Refer to SOP PDP-LABOP-01, subsection 5.1.

6.2 Sample Preparation

Mechanically homogenize each tissue until a visually homogeneous mixture is attained. The laboratory shall use its discretion in the utilization of dry ice during the homogenization procedure. The laboratory shall follow the procedures in SOP PDP-LABOP-01 sections 5.2 and 5.3 for the storage of homogenates and extracts.

6.3 Sample Storage

An adequate portion of the homogenized tissue shall be held in reserve in case re-analysis is required. This portion shall be distributed among several small containers (polypropylene or styrofoam recommended) rather than one large container and stored at 0°C or less. The laboratory's internal documentation shall specify or define an "adequate portion" and distribution.

6.4 Sample Analysis

6.4.a Weighing of Analytical Portion

An appropriate amount (i.e., weight) of homogenized tissue for sample analysis shall be defined in an internal laboratory SOP. The internal SOP shall also define the level of precision associated with the weight. The specified level of precision shall not be more than ± 0.05 grams for a 10-gram sample, and ± 0.10 grams for a 20-gram sample.

6.4.b Sample Set Requirements

6.4.b.1 A sample set is the group of test portions that are spiked with the designated process control and extracted on a single day along with the required QC samples. Each set shall

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consist of no more than 20 analytical samples. Required QC samples per set include a reagent blank, matrix blank, and matrix spike(s).

6.4.b.2 The matrix spike(s) shall be prepared at approximately 2xLOQ with the identified marker pesticides. Refer to SOP PDP-QC-13 section 5.2 for marker pesticide requirements.

6.4.b.3 Each analytical sample shall be spiked with the appropriate process controls at approximately 5xLOQ. Refer to SOP PDP-QC-13 section 5.3 for process controls requirements. All components of sample sets shall be subject to the entire analytical test process as detailed in approved methodology.

6.4.c Analytical Requirements

6.4.c.1 Each tissue shall be analyzed for compounds as specified in the applicable SOP PDP-QC-13 addendum.

6.4.c.2 Some analytical reference materials are not readily available from commercial sources; therefore, materials may be requested from the US EPA Standards Repository. Please contact Chuck Stafford, EPA, Fort Meade, MD, at 410-305-3091 or 410-305-2914 (phone) or 410-305-2999 (fax). Other compounds may also be requested from the EPA Standards Repository, if desired.

6.4.c.3 Use of alternate suppliers whose reference material fails to meet the minimum requirements outlined in SOP PDP-STD-01 must be approved by the PDP Technical Director and documented.

6.4.d Method Validation Requirements:

All compounds shall undergo validation requirements according to SOP PDP-QC-07. Refer to SOP PDP-QC-13 section 5.3 for commodity grouping information.

6.5 Data Reporting

6.5.a Reportable Residues

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If a sample contains a residue concentration \$LOQ a reportable residue has occurred. The Reportable Residue Form for Animal Tissues (Attachment 2) shall be filled out and emailed or faxed immediately to MPO and USDA, FSIS. The USDA, FSIS contacts are Dr. Doritza Pagan-Rodriguez and Margaret O’Keefe. In addition, the form shall be emailed to the FSIS residue mailbox (residue@fsis.usda.gov)

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6.5.b Presumptive Tolerance Violations (PTVs)

Reporting requirements, as specified in SOP PDP-DATA-02, subsection 5.3 do not apply.

6.5.c All data (manual or electronic) shall be transmitted according to established procedures. The laboratory shall report all results to MPO; the USDA, FSIS designee(s); and the FSIS residue mailbox each Friday for samples that were received by Wednesday. Samples received after Wednesday will be held and analyzed with the next week’s samples. The laboratory shall notify MPO; the USDA, FSIS designee(s); and the FSIS residue mailbox if results will be reported late due to instrument problems.

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6.6 Sample Disposal

Stored homogenates shall be disposed of when all requirements for acceptability criteria have been met and results have been successfully transmitted to MPO. Disposal shall be documented (e.g., freezer log, sample log, extraction worksheet, RDE chain-of-custody inputs) and shall contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.

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02/22/06

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02/27/06

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*ORIGINAL SIGNATURE PAGE MAINTAINED BY USDA, AMS, SCIENCE & TECHNOLOGY, MONITORING PROGRAMS OFFICE
ELECTRONICALLY REPRODUCED SIGNATURES*

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Revision 5

February 2006

Monitoring Programs Office

- Updated to include current reporting requirements

<i>Internal lab code here</i>		U.S. DEPARTMENT OF AGRICULTURE FOOD SAFETY AND INSPECTION SERVICE REQUESTED SAMPLE PROGRAMS <input type="checkbox"/> FOOD CHEMISTRY <input type="checkbox"/> MICROBIOLOGY <input type="checkbox"/> RESIDUE			<i>Barcode here</i> 1. SAMPLE FORM NO.		
PART I. SAMPLE COLLECTION AND MAILING INSTRUCTIONS							
2. SAMPLE TYPE CODE	3. EST. NO.	4. COLLECT TISSUES/SAMPLES ON			5. REGION/ DISTRICT	6. STATE	7. CIRCUIT/IFO
		Day of:	Week of:	Within 30 days of:			
8. ESTABLISHMENT ADDRESS/SAMPLE COLLECTION ADDRESS (i.e., Est., Retail Store)				9. NAME & ADDRESS OF RECEIVING LABORATORY			
10. SLAUGHTER CLASS CODE	11. SPECIES TO COLLECT	12. TISSUE	13. ANALYSIS REQUESTED				
14. PROJECT NO.	15. COUNTRY OF ORIGIN		16. COUNTRY CODE		17. FOREIGN EST. NO.		
18. ADDITIONAL INSTRUCTIONS							
PART II. COLLECT SAMPLE INFORMATION (To be completed by sample collector)							
19. DATE COLLECTED	20. DATE SENT TO THE LAB	21. PRODUCT TEMPERATURE			22. PRODUCT HELD <div style="text-align: right;"><input type="checkbox"/> Yes <input type="checkbox"/> No</div>		
23. FSIS 9540-1 NO.	24. LOT NO.	25. IMPORTS <div style="text-align: right;"><input type="checkbox"/> Normal (06) <input type="checkbox"/> Increased (07) <input type="checkbox"/> Special (53) <input type="checkbox"/> Hold (24)</div>					
26. PRODUCER/DEALER/OWNER-NAME/ADDRESS/STATE/ZIP CODE					27. ANIMAL ID (Tag No.)		
28. REMARKS							
29. COLLECTOR'S SIGNATURE		30. NAME OF COLLECTOR (Print)		31. BADGE NO.	32. TELEPHONE NO. AT EST.		
33. IF THE REQUESTED SAMPLE(S) ARE NOT COLLECTED, CHECK OFF THE APPROPRIATE REASON & RETURN THIS FORM TO THE LAB INDICATED ABOVE. (72) <input type="checkbox"/> REQUESTED PRODUCT(S) NOT PRODUCED DURING THE SAMPLING TIME FRAME (If checked, plant wil be subject to sampling at a later date.) (60) <input type="checkbox"/> PLANT DOES NOT SLAUGHTER SPECIES/CLASS OR PRODUCE THE REQUESTED PRODUCTS (If checked, plant will be removed from this sampling program.) (67) <input type="checkbox"/> NEEDED SUPPLIES OR APPROPRIATE SHIPPING CONTAINER NOT AVAILABLE. (53) <input type="checkbox"/> OTHER (Explain):							
PART III. LABORATORY RECEIPT INFORMATION							
34. SAMPLE PACKAGING <div style="display: flex; justify-content: space-around;"> <input type="checkbox"/> 3034 Intact Package <input type="checkbox"/> 3035 Non-Intact Package </div>					35. SAMPLE RECEIPT DATE		
36. PRODUCT CODE		37. NO. SAMPLES IN COMPOSITE		38. SAMPLE RECEIPT TEMPERATURE			
39. SAMPLE RECEIPT CONDITION CODE		40. SEAL CONDITION CODE		41. DISCARD CONDITION CODE			

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**SOP PDP-LABOP-12, Revision 5
Attachment 2
Reportable Residue Form for Animal Tissue**



United States
Department of
Agriculture

Agricultural
Marketing
Service

Science & Technology
Field Laboratory Services

National Science Laboratory
801 Summit Crossing Place, Suite B
Gastonia, NC 28054

(704)867-3873 VOICE
(704)853-2800 FAX

REPORT OF SAMPLE RESULTS

Reporting Information

Margaret O'Keefe
USDA/FSIS/OPHS/ZDRSD
Mail Drop Room 343 Aerospace Center
1400 Independence Ave., SW
Washington, DC 20250-3700

FSIS Form #:

Establishment #:

Establishment Address

Lot #:

PDP ID #:

Internal Lab #:

LIMS ID #:

Date Collected:

Product Description:

Producer Address

Date Shipped:

Name of Collector:

Date Received:

Collector Phone #:

Date Completed:

ANALYTICAL TEST RESULTS

Analyte Name

Results (ppb)

Quantitation Limit (ppb)

SIGNATURE OF APPROVING OFFICIAL

WARNING: Any person who knowingly falsely makes, issues, alters, forges, or counterfeits this report, or participates in any such actions, is subject to a fine of not more than \$1,000 or imprisonment for not more than one year, or both (7 U.S.C. 1662 (h)). The information contained within this report of analytical test results is applicable only to the materials identified within and is, to the best of our ability and knowledge, accurate with regard to the client's specifications. The laboratory shall not be responsible for errors due to the client's failure to provide information critical to the currency of contract specifications, etc. The report is receivable in all courts of the United States as prima facie evidence of the truth of the statements therein contained. It does not excuse failure to comply with any applicable Federal or State Laws.